

Polyimide Composites Based on Asymmetric Dianhydrides (a-ODPA vs a-BPDA)

Kathy C. Chuang

NASA Glenn Research Center, Cleveland, OH 44135
(216) 433-3227; Kathy.Chuang@grc.nasa.gov

Jim M. Criss

M & P Technologies, Inc. Marietta, GA 30314
(770) 652-7638; jim.m.criss@mandptechnologies.com

Eric A. Mintz

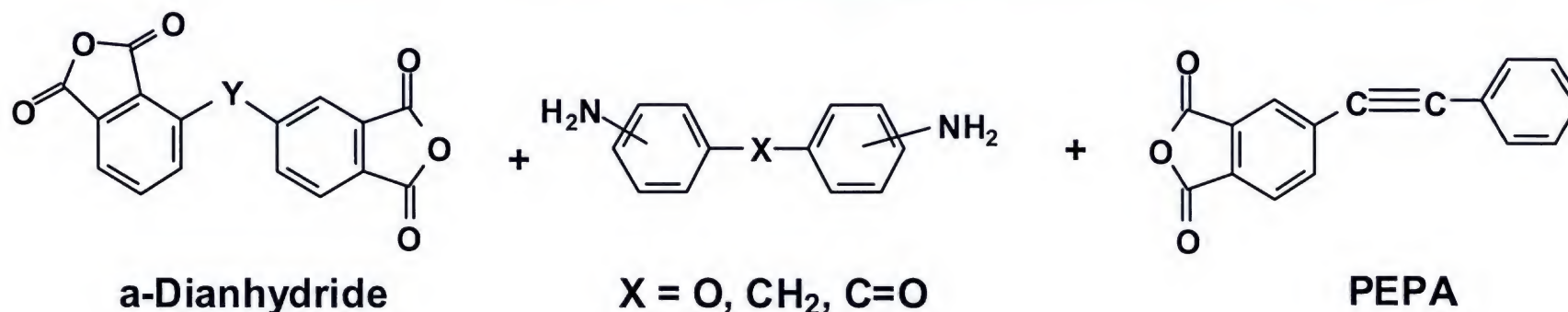
Center for High Performance Polymers and Composites,
Clark Atlanta University, Atlanta, GA 30314

High Temperature Polyimide Composites

Materials and Processing

- ♦ PMR-15, PMR-II-50, AFR-PE4, PETI-5 composites all require solvent-based prepregs for processing
⇒ *time consuming, costly and hazardous*
- ♦ Fabricate polymer composites via RTM process
⇒ *produce 30% cost saving for complex parts*
- ♦ a-BPDA based imide resins have shown to exhibit low-melt viscosity (10-30 poise) at 280 °C
 - *amenable to low-cost RTM process*
 - *advance PMC temperature capability to 260-315°C beyond state-of-the-art RTM resins, such as epoxy (177 °C) & BMI (232 °C)*

Low-melt Viscosity Imide Resins



Imidized Oligomers

Advantages of imide resins containing α -dianhydrides

- *Lower melt viscosities*
- *Higher T_g 's*

Objectives

- ◆ **Prepare imide resins with low-melt viscosities (~10 poise) for RTM or VARTM using asymmetric dianhydrides, namely α -ODPA and α -BPDA**
- ◆ **Process low-melt viscosity imide resins by RTM into composite panels**
- ◆ **Evaluate mechanical properties at 288-315°C (550-600°F)**

Physical Properties of Imide Oligomers/ Resins Based on a-BPDA and 4-PEPA

Resin	Diamine	Oligomer Min. η @280 °C by Brookfield ¹ (Poise)	Oligomer Min. Complex [η] [*] @280°C ² (Poise)	Cured Resin T_g (°C) NPC ³ byTMA	Cured Resin T_g (°C) PC ⁴ @ 650°F By TMA ⁵
RTM370	3,4' -ODA	8.8	6.5	342	370
RTM350	3,4' -MDA	7.4	20	338	350
RTM330	3,3' -MDA	1.5	10	288	330

¹ Absolute viscosity measured by Brookfield Viscometer at 280 °C.

² Complex viscosity measured by Aries Rheometer, using parallel plates.

³ NPC = No Post cure

⁴ PC = Post cured at 343 °C (650 °F) for 16 hrs.

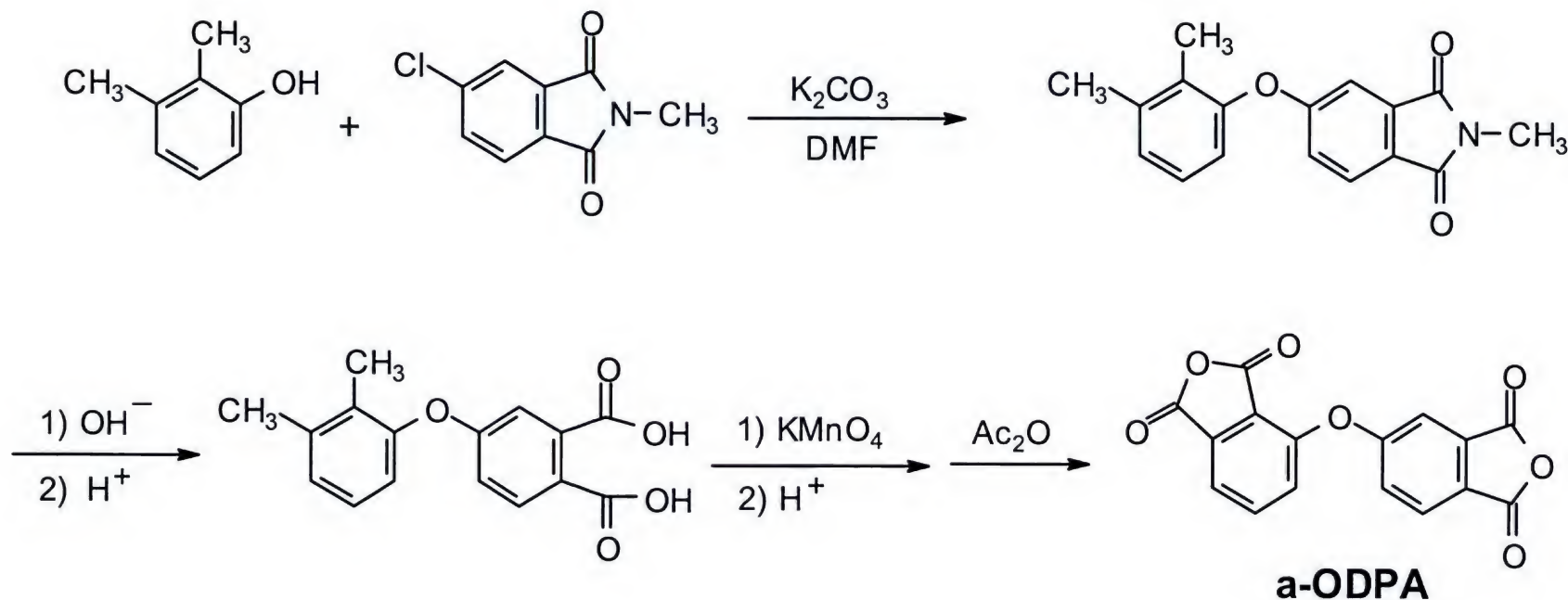
⁵ TMA =Thermal mechanical analysis heated at 10 °C/min, using expansion mode.

3,4' -ODA = 3,4' -Oxydianiline

3,4' -MDA = 3,4' -Methylenedianiline

3,3' -MDA = 3,3' -Methylenedianiline

a-ODPA Synthesis



Q. Li, X. Fang, Z. Wang, L. Gao, M. Ding, J. Polymer Science, Part A: Polymer Chemistry, **41**, 3249, (2003)

Physical Properties of Imide Oligomers/Resins Based on a-ODPA

Dianhydride	Diamine	Oligomer Min. η @280 °C by Brookfield ¹ (Poise)	Oligomer Min. Complex [η]*@260°C ² (Poise)	Cured Resin T _g (°C) NPC ³ By TMA	Cured Resin T _g (°C) PC ⁴ @ 650°F By TMA ⁵
a-ODPA	3,4'-ODA	3.5	15.0	296	329
a-ODPA	3,4'-MDA	4.0	14.0	270	294 ⁶
a-ODPA	3,3'-MDA	2.5	3.0	273	266 ⁶
a-ODPA	3,3'-DABP	3.0	4.0	270	297

3,3'-DABP = 3,3'-Diaminobenzophenone

¹ Absolute viscosity measured by Brookfield Viscometer at 280 °C.

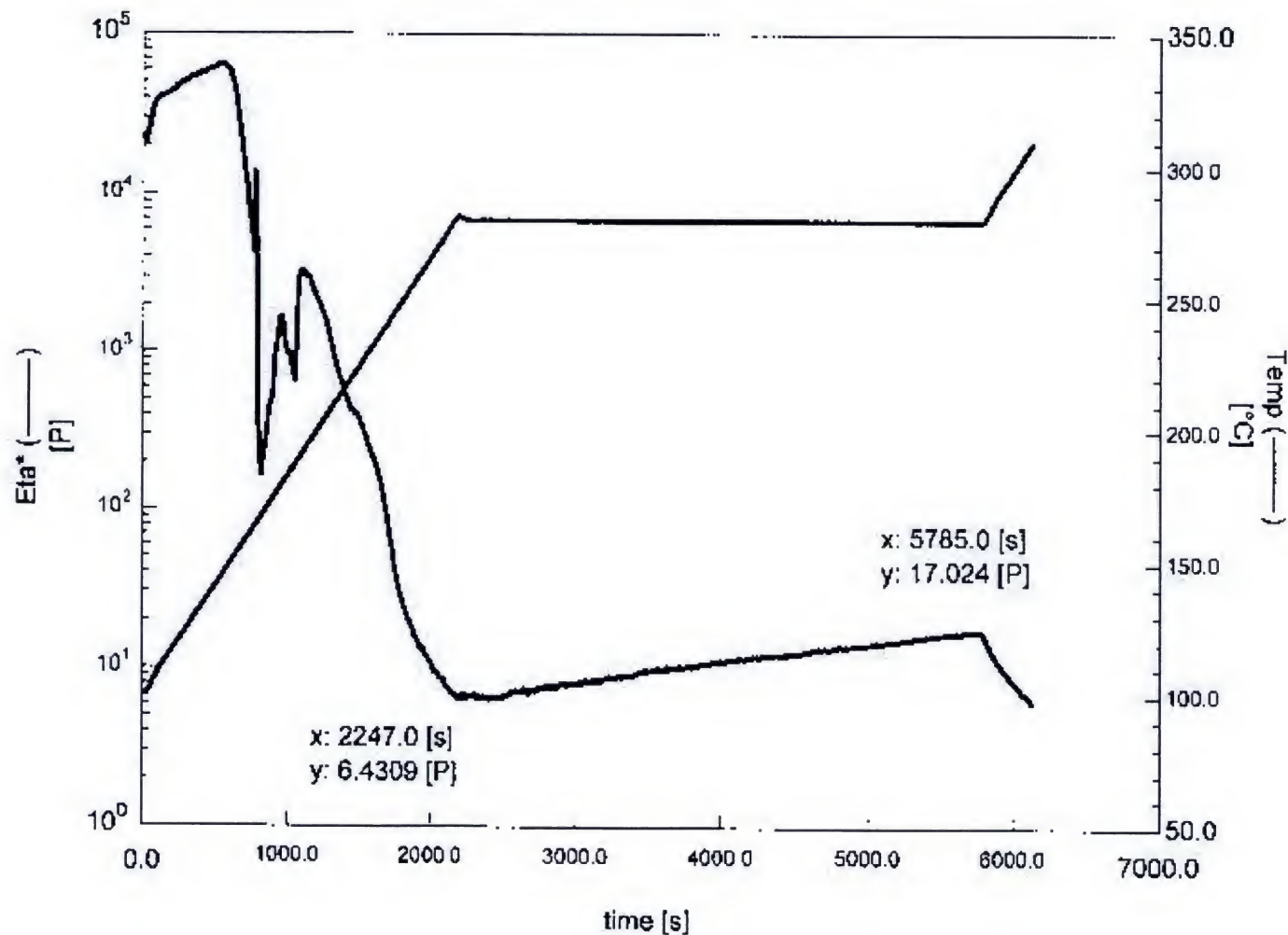
² Complex viscosity measured by Aries Rheometer, using parallel plates.

³ NPC = No Post cure

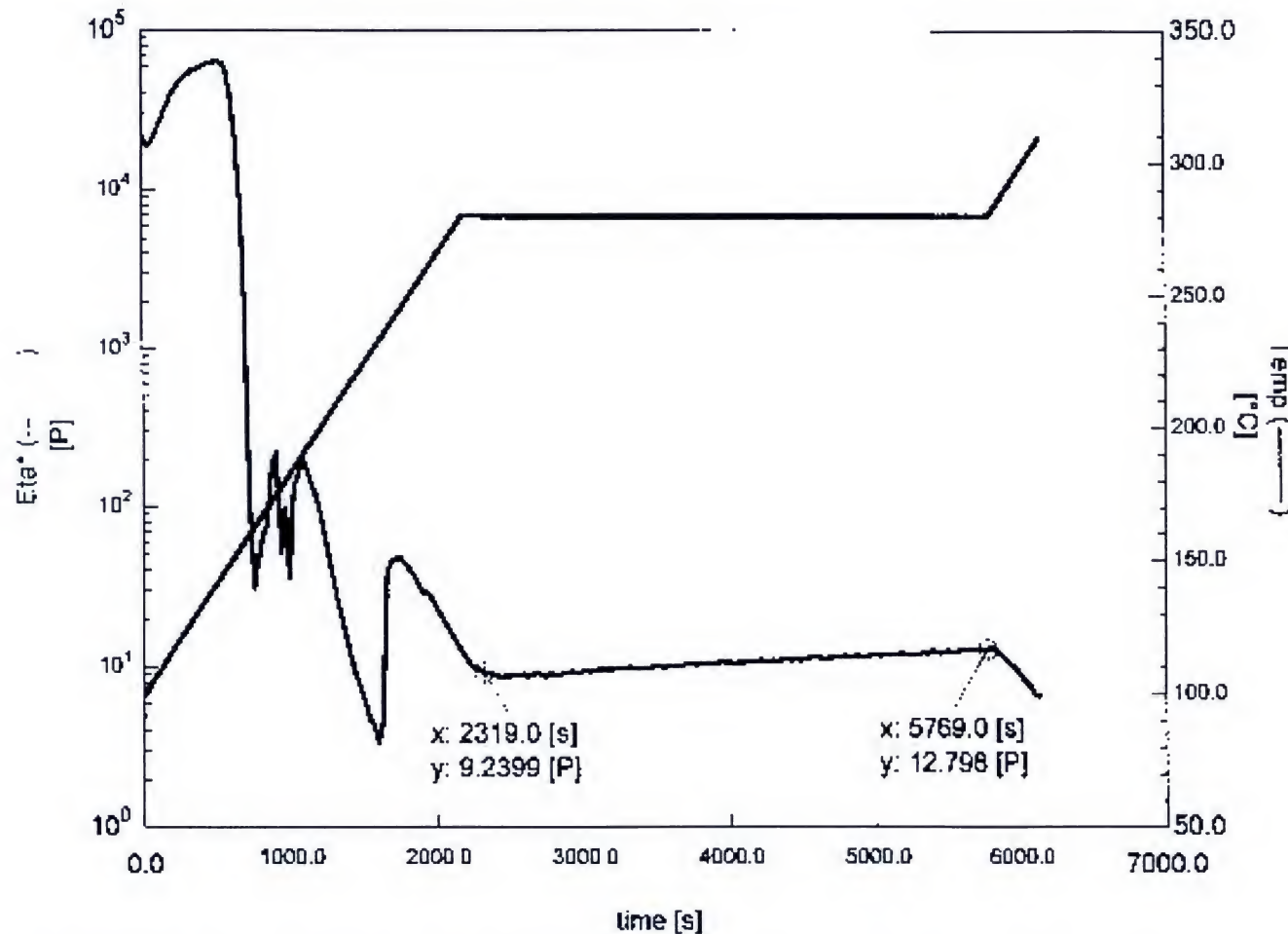
⁴ PC = Post cured at 343 °C (650 °F) for 16 hrs.

⁵ TMA = Thermal mechanical analysis heated at 10 °C/min, using expansion mode

Rheology of a-BPDA/3,4'-ODA/PEPA Imide Resins at 280 °C Hold

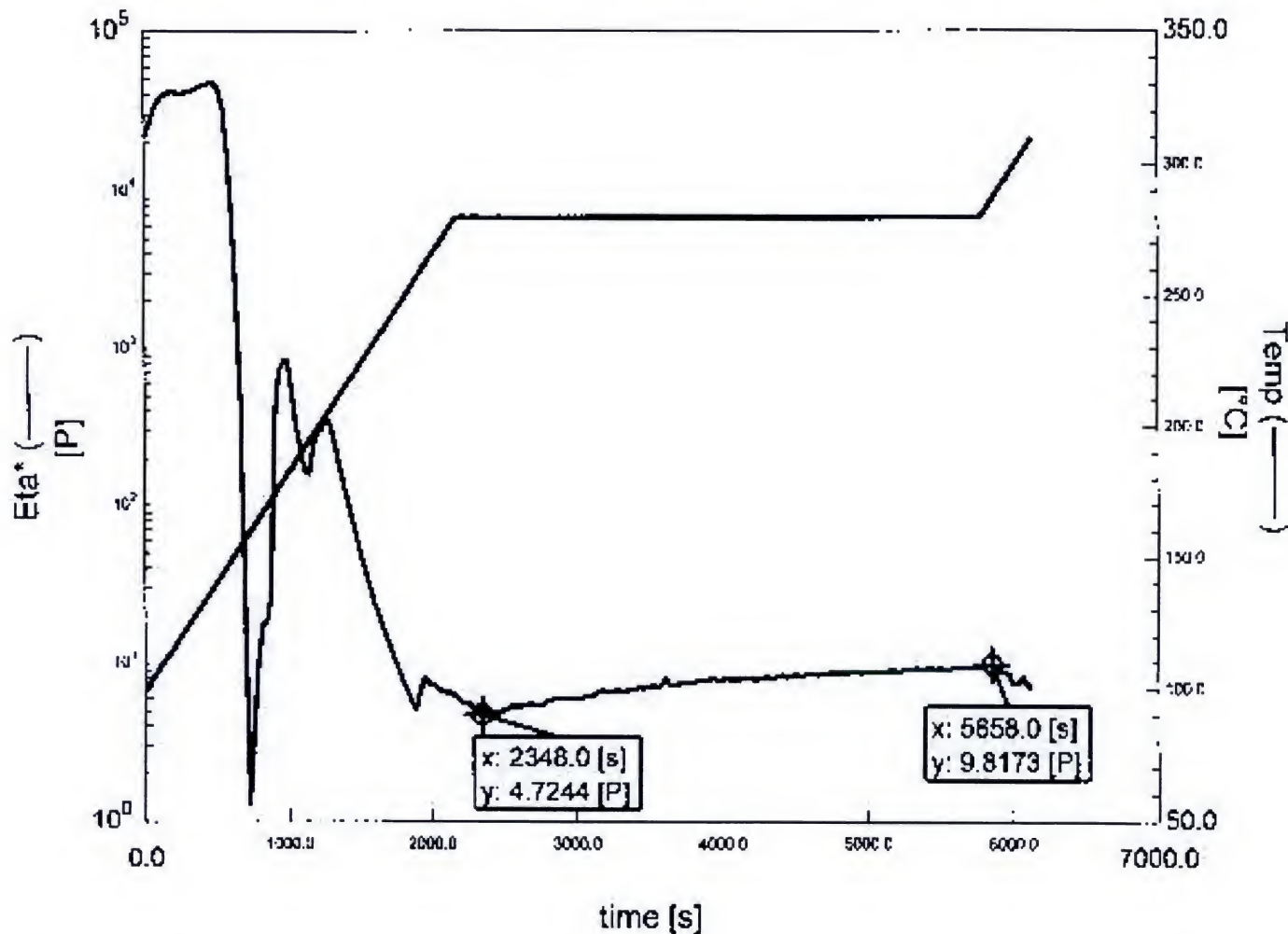


Rheology of a-ODPA/3,4'-ODA/PEPA Imide Resins at 280 °C Hold



Maintained low-melt viscosity (9-13 poise) at 280 °C

Rheology of α -ODPA/3,3'-DABP/PEPA Imide Resins at 280 °C Hold

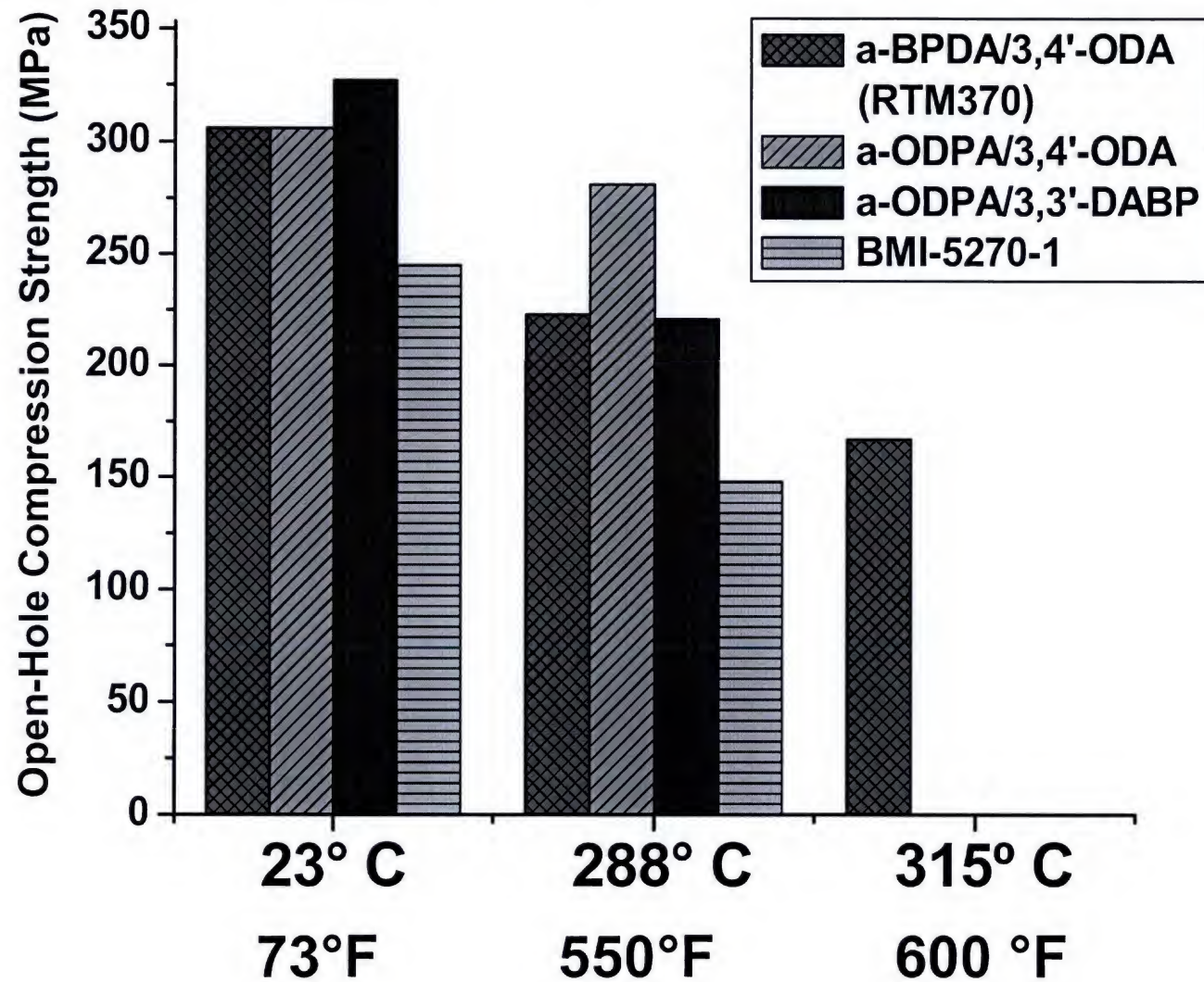


Maintained low-melt viscosity (4-10 poise) at 280 °C

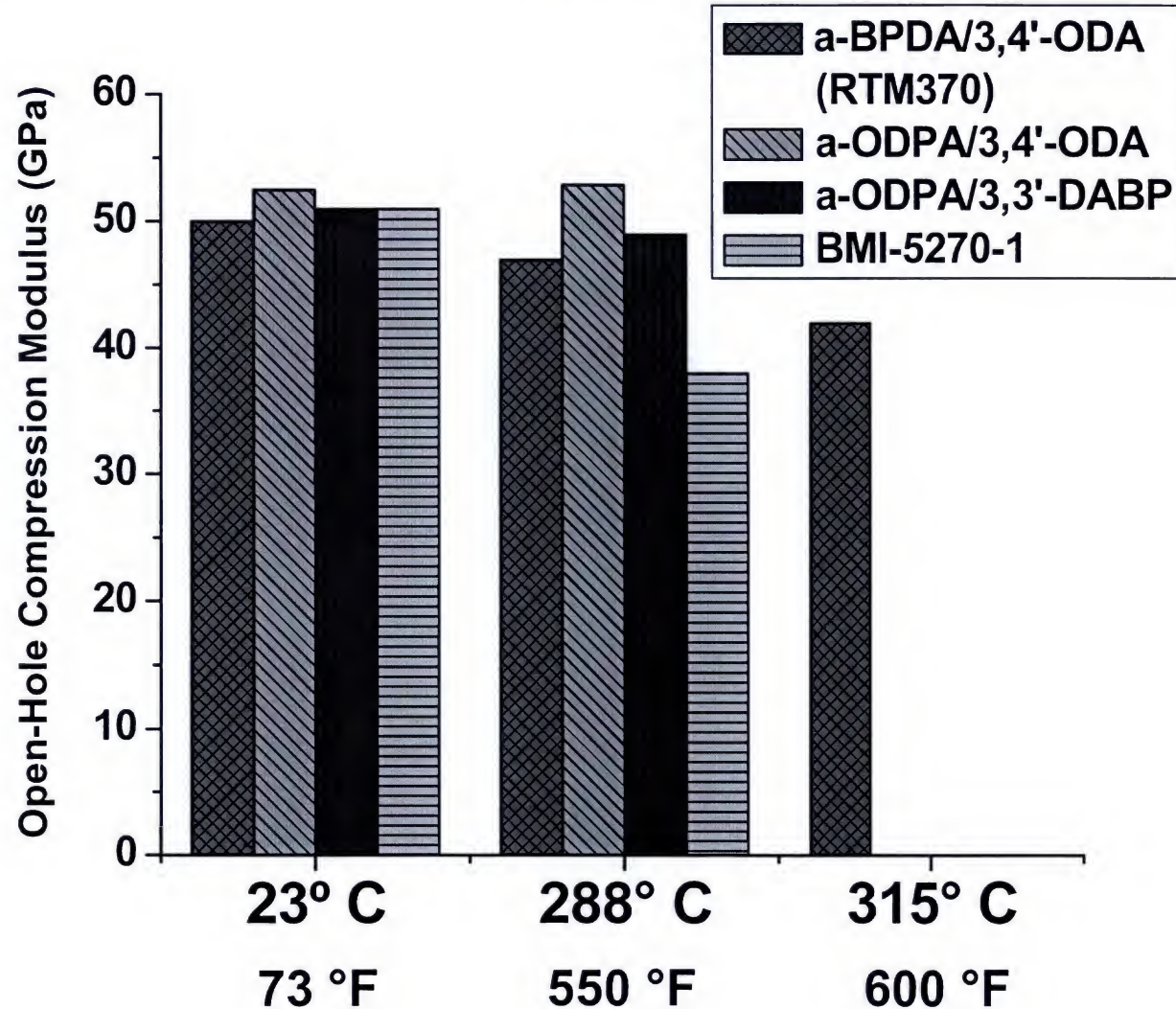
Mechanical Properties of RTM Resins Based on a-BPDA and a-ODPA

Property	Test Temp. (°C)	RTM370 a-BPDA 3,4'-ODA 4-PEPA	a-ODPA 3,4'-ODA 4-PEPA	a-ODPA 3,3'-DABP 4-PEPA	BMI-5270-1
Open-Hole Compression Strength (MPa)	23	306	306	327	245
	288	223	281	221	148
	315	166	---	---	---
Open-Hole Compression Modulus (MPa)	23	50	51	53	51
	288	47	49	53	38
	315	42	--	--	--
Short Beam Shear Strength (MPa)	23	62	70	58	37
	288	43	33	36	15
	315	32	--	--	--

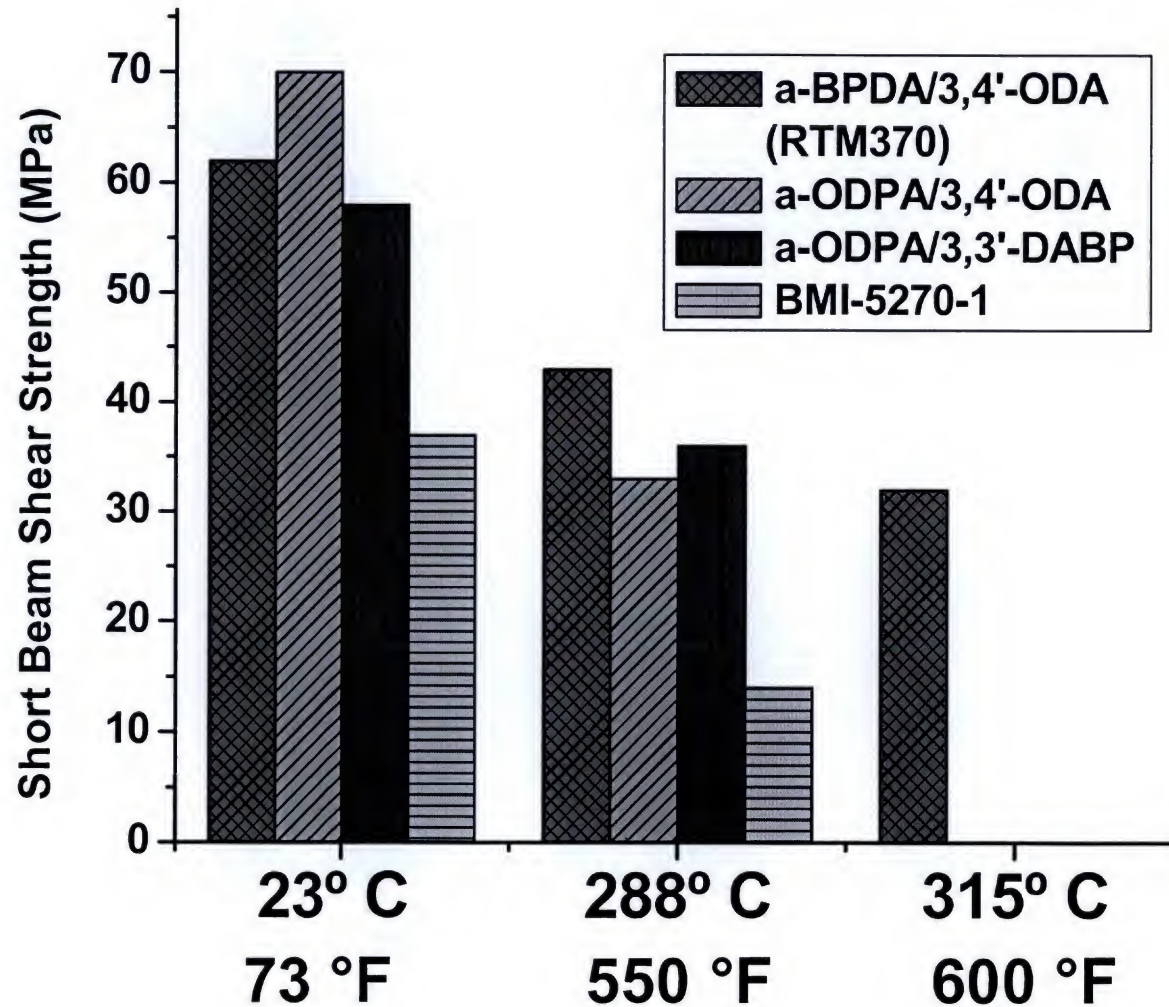
Open-Hole Compression Strength of T650-35 a-BPDA & a-ODPA Composites vs BMI-5270-1



Open-Hole Compression Modulus of T650-35 a-BPDA & a-ODPA Composites vs BMI-5270-1



Short Beam Shear Strength of T650-35 a-BPDA & a-ODPA Composites vs BMI-5270-1



Summary

- ◆ **RTM Resins based on a-ODPA and a-BPDA with kinked diamines exhibit low-melt viscosity (~ 10 poise)**
- ◆ **Composites made from a-ODPA resins ($T_g = 265-330^\circ\text{C}$) by RTM display good mechanical properties at 288°C (550°F), but soften at 315°C (600°F)**
- ◆ **Composites of RTM370 based on a-BPDA retain excellent mechanical properties at 315°C , exceeding BMI-5270-1 capability**

Acknowledgements

- ◆ **Funding from AFOSR: Dr. Charles Lee**
- ◆ **NASA Supersonic Program**
- ◆ Dan Scheiman (ASRC): Thermal Analysis
- ◆ Linda McCorkle (OAI) : Rheology
- ◆ Baochau Nguyen (OAI): Synthesis Modification
- ◆ Brian Shonkwiler(CAU): Mechanical Testing